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# New Experimental-Technique For The Fabrication Of Ion Diffused Optical-Glass Wave-Guides

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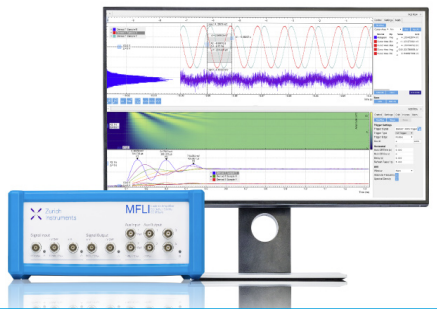

# New experimental technique for the fabrication of ion diffused optical glass waveguides

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A new electric-field-assisted ion diffusion technique has been used for the fabrication of optical waveguides in glass. The construction of the setup is discussed in detail. Ion exchange in standard glass slide can be performed very efficiently and the setup can be conveniently used for double diffusions which are required for the fabrication of buried waveguides. Silver ions of various mole fractions were diffused in glass by this technique. Diffusion depth measurements were made both for the fabricated planar and channel waveguides.

## I. INTRODUCTION

Ion exchange has been the most popular technique to produce waveguides in glass substrates.[1,2] The process involves the exchange of monovalent ions of larger electronic polarizability such as $K^+$, $Ag^+$, $Tl^+$, and $Cs^+$ with an ion of smaller polarizability such as $Na^+$, which is a usual component of the soda-lime or borosilicate glass.[2] Any change in the composition of a glass produces a refractive index change because of alteration of the electronic structure of the material. The ion exchange process creates an increase in the refractive index thus forming a guiding layer at the surface of the glass substrates. There has been considerable interest in the study of these glass waveguides because of their potential applications in integrated optical components like star couplers, multiplexers, and demultiplexers.[3–6] These low-cost glass waveguides are very much compatible with optical fibers. They have low propagation loss and can be integrated with the optical systems.[7] Though titanium-diffused $LiNbO_3$ low-loss optical waveguides are strong candidates for single-mode fiber optical applications,[8] they are not suitable for multimode applications where one needs deep diffused channel waveguides. Deep channel waveguides of the order of 50–100 $\mu$m can be easily achieved in glass.[9,10] These deep diffusions are achieved by the application of an electric field during the thermal diffusion process. The application of an electric field provides the additional force during the exchanged ions at a faster pace, thus reducing the process time as compared to thermal diffusion alone. In the case of Ti-diffused $LiNbO_3$ waveguides, electric field cannot be applied to enhance the diffusion process. Because of this, waveguides with depths greater than 2 $\mu$m are difficult to achieve and it is time consuming. Hence glass waveguides are very attractive for multimode fiber optic applications. Silver ions can be introduced into glass from molten silver-nitrate ($AgNO_3$) solution[11,12] or through silver films previously vacuum deposited[9,13,14] at elevated temperature in the range of 250–300 °C. $Ag^+$ diffusion from silver film is not an attractive technique because the diffusion approaches a Gaussian function as it behaves like a limited source diffusion.[12] When the diffusion occurs from a bath of constant concentration as in the case of $AgNO_3$, the diffusion is very nearly complementary error function. In the $AgNO_3$ process the refractive index can be easily adjusted below the saturation level by diluting the silver salt with sodium nitrate ($NaNO_3$).[15]

In most of the previous field-assisted $Ag^+$ diffusion processes using $AgNO_3$, one side of the glass slide touches the molten solution and the other side has a metal film (Au, Cr-Au, Al, or Ag) which acts as cathode. The metallic back electrode tends to deteriorate with time due to the reactive $Na^+$ accumulating on the cathode side.[16] Further, during the diffusion process, because of the thin (1 mm) nature of the commercially available glass substrates being used for the fabrication of the waveguides, there is chance of molten salt spilling or spreading on the back metal electrode causing short circuit. Lilienhof *et al.*, using gold film as the cathode metal, aligned the surface of the sample to the surface of the melt with the aid of an adjusting laser.[10] In their system the ion current was fed into the control unit which automatically adjusted the vertical position in order to avoid the short circuit. Lagu and Ramaswamy used an electrolytic release of silver ions from a silver rod electrode immersed in the molten $NaNO_3$.[17] By controlling the current, the silver concentration in the salt, and therefore the rate of $Ag^+$-$Na^+$ ion exchange, was controlled, yielding the desired index change. The exchange carried out at 300 °C for 120 min yielded low-loss waveguides with only two modes with an index increase $\Delta n = 0.005$. Recently, Chao and Lee used a new technique to fabricate low-loss waveguides in BK-7 glass.[18] In this method, a quartz crucible contained the molten salts. The glass substrate, using a polyimide as a sealant, separated this crucible into two compartments one side containing $NaNO_3$ and the other side containing the mixture of $AgNO_3$ and $NaNO_3$. Aluminum plates immersed in the molten salt solutions served as the electrodes. Diffusion carried out at 320 °C using 0.5–1.0 mol % of $AgNO_3$ yielded low-loss (0.1–0.4 dB/cm at 632.8 nm) waveguides. This method is also is not very convenient because every time a sample is to be diffused it has to be sealed with polyimide in the groove and improper sealing will result in the short circuiting of the solutions on both sides.

Buried layers are necessary in guided structures to obtain suitable index profile symmetry and low propagation

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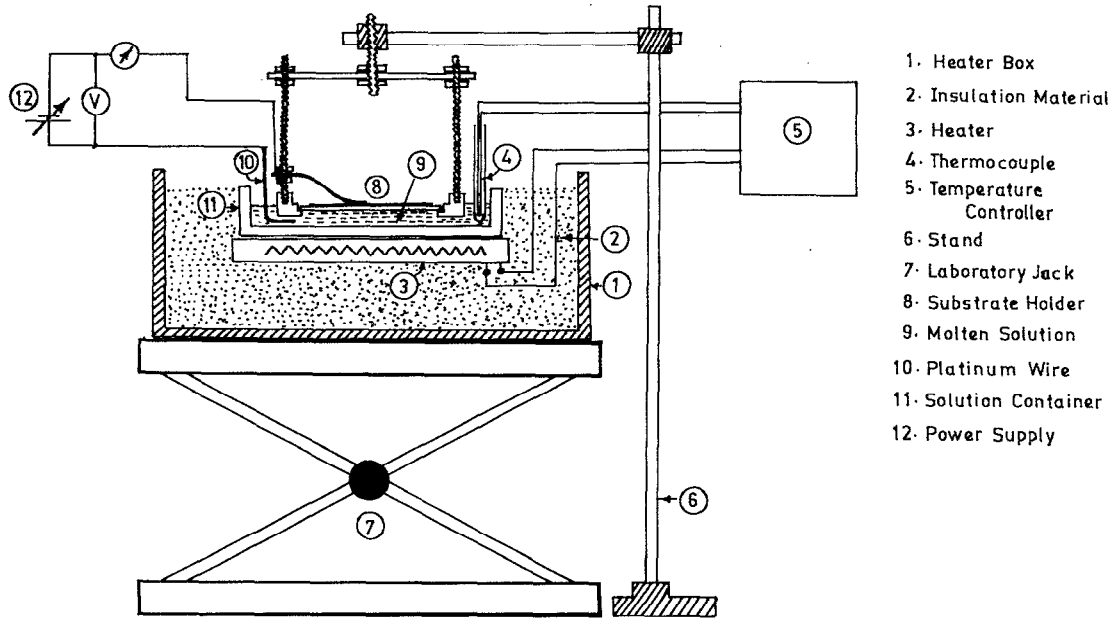


FIG. 1. Schematic of the experimental setup.

loss.[2] The profile symmetry maximizes coupling between waveguides and fibers whose profile is symmetric. The loss due to surface roughness is reduced in buried guided layers as they are below the substrate surface. In the ion exchange process buried layers can be produced by a double-diffusion method.[3,12,19,20] The first diffusion process introduces ions that increase the refractive index. This is followed by the second diffusion where the introduced ions lowers the refractive index in the vicinity of the surface yielding a graded-bell-shaped profile. For multimode buried layer waveguide fabrication electric-field-assisted double diffusion is the most compatible technique.

In this work a new experimental technique was designed to achieve the exchange of ions in glass by the application of an electric field. The setup is quite simple to realize and diffusion of the species can be performed easily. This new technique overcomes many difficulties seen by previous workers for the fabrication of multimode waveguides. The setup was used to diffuse $Ag^+$ ions into glass substrates. Various silver mole concentrations with $NaNO_3$ were tried and diffusion depths were measured for the fabricated planar as well as channel waveguides.

## II. EXPERIMENT

The schematic of the diffusion setup is shown in Fig. 1. The setup consists of mainly three major parts, namely, (a) heater box, (b) molten salt container, and (c) substrate holder assembly. The construction of these parts is described as follows.

### A. Heater box

An aluminum box (20 cm×15 cm×12.5 cm) was packed with thermal insulation to a height of about 8 cm. A 400 W strip heater (14 cm×6.5 cm) (Chromalox) was

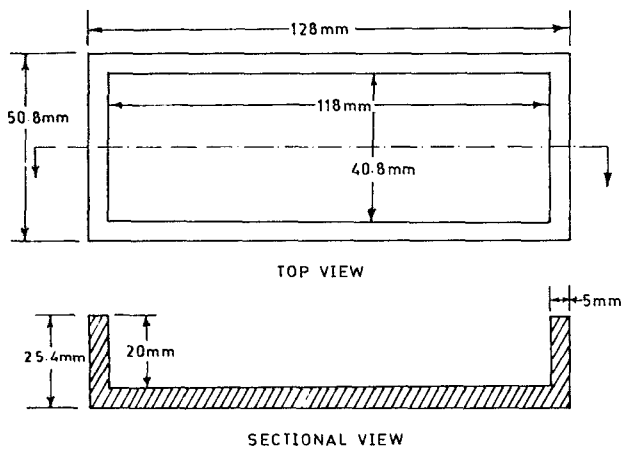


FIG. 2. Schematic of the solution container.

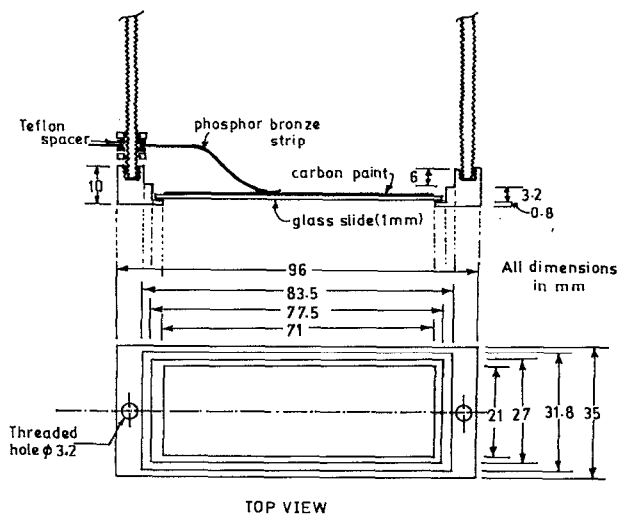


FIG. 3. Schematic of the substrate holder.

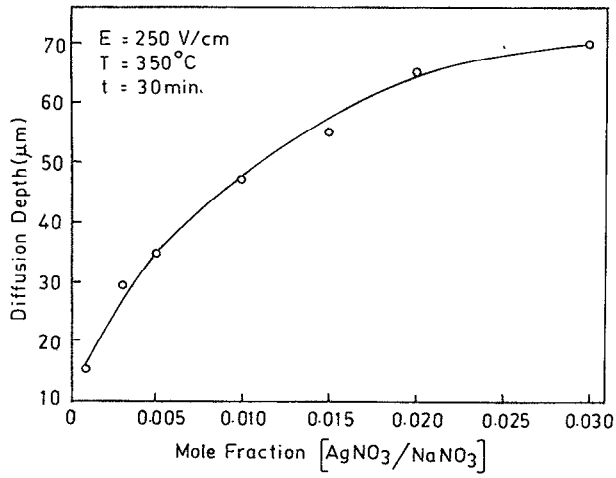


FIG. 4. Variation of diffusion depths with silver mole fractions.

placed on the packed insulation. The strip heater was powered by a temperature controller (Omega Model 1001). A *K*-type chromel-alumel thermocouple, inserted inside a glass tube, served as the temperature sensor in the molten bath. The thermocouple leads were connected to the temperature controller. The entire heater assembly was placed on a laboratory jack whose height could be adjusted very precisely.

### B. Molten salt container

The molten salt container was in the shape of a rectangular box and the schematic with dimensions is shown in Fig. 2. A 2.5-cm-thick aluminum block was the starting material and the rectangular boat was carved out using a milling machine. This was followed by complete anodization of the container surface. The salt container was placed on the top of the heater and was packed with the insulation material on the sides.

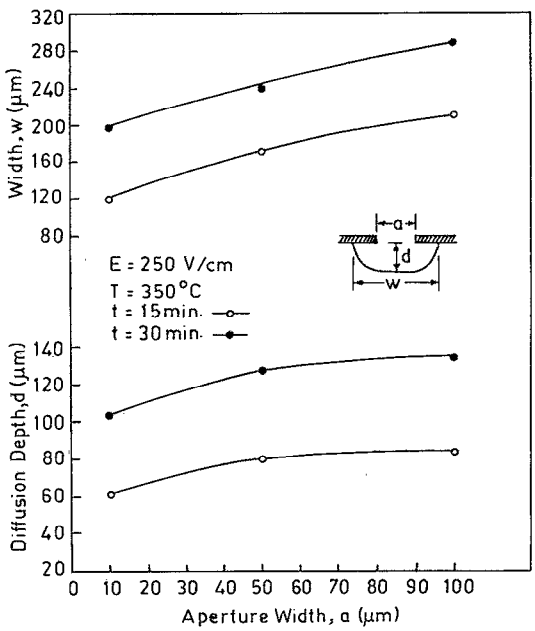


FIG. 5. Variation of diffusion depths and widths with mask aperture widths.

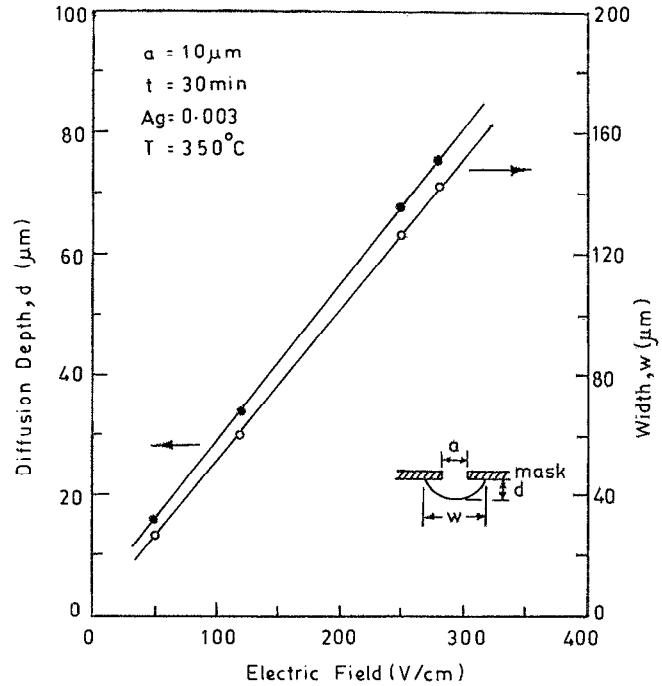


FIG. 6. Variation of diffusion depths and widths with electric field.

### C. Substrate holder assembly

The schematic of the substrate holder with dimensions is shown in Fig. 3. The starting material was 1.25-cm-thick aluminum plate. The required shape with slot and groove was carved out using the milling machine. The sample holder was completely anodized. Stainless-steel threaded rods (diameter 3.2 mm) were used to hold the substrate holder. The entire assembly was fixed to a laboratory stand whose position could be adjusted to any convenient height. A phosphor-bronze strip (1.25 cm wide) was bent in the required shape and was attached to one of the stainless-steel rods with the help of nuts. Teflon spacers were used to insulate the strip from the rod. A standard glass substrate (75 mm×2.5 mm×1 mm) could be placed in the groove of the substrate holder and could be held firmly by adjusting the tension on the phosphor-bronze strip with the help of nuts.

### D. Ion exchange diffusion

Corning sodalime glass slides (2947) were used for all diffusion experiments. The glass slides were thoroughly cleaned using acid and solvents. For the fabrication of channel waveguides, a 300-nm-thick aluminum film was vacuum deposited and patterned using the required mask by photolithography. Aluminum film served as excellent masks for silver ion diffusion. Initially, a 300-nm-thick Cr-Al or Cr-Au was tried as back electrode on the glass for diffusion. The deterioration of the electrode was observed within 10 min of diffusion for the typical current densities

used in the experiments. This problem of electrode deterioration was overcome by coating the backside with carbon paint (SPI Supplies). The paint dried within a few minutes. One application of carbon paint by brush gave nearly a 50–100 $\mu$m thick electrode. This thick metal electrode cannot be achieved by the conventional vacuum deposition technique and is time consuming. This carbon paint served as excellent cathode contact with glass. No deterioration of the carbon electrode was observed even after 60 min of diffusion for this thick electrode. Thicker paint could be applied if one needs to carry out diffusions for longer periods. The glass slide was placed on the substrate holder groove and the tension on the phosphor-bronze strip was adjusted to have good contact with carbon paint cathode. A $AgNO_3$-$NaNO_3$ mixture of the required molar concentration was initially melted and the temperature was set at 350 °C by adjusting the temperature controller. All diffusions were carried out at this temperature. Sometimes, the sudden contact of the glass slide with the molten liquid resulting in cracking due to thermal shock. To prevent this, the following procedure was adopted. Initially the substrate holder with slide was kept at 4 cm above the liquid surface. After waiting for three minutes the jack (with heater box) was slowly raised so that the gap between the slide and the liquid surface was reduced by 1 cm. This was continued until the slide was positioned at about 2–3 mm above the liquid surface. After reaching this position, the slide was allowed to touch the molten liquid surface. This procedure was very successful and even after carrying out hundreds of diffusions, not a single sample cracked. Once the liquid wets the surface it spreads uniformly. If any bubble was observed under the slide, the glass slide was raised by adjusting the jack and subsequently immersed. Once it was made sure that no bubbles were trapped below, the substrate holder was raised to about 1 mm and the surface tension of the liquid kept the slide in contact with the molten liquid. This prevented any possible leakage of the liquid to the back electrode during diffusion.

Platinum wire immersed in the liquid served as the anode lead for the power supply. The required voltage and current were adjusted and the ion diffusion was initiated. After diffusion was completed for a specific period, the glass slide was quickly withdrawn and kept about 4–5 cm above the liquid surface. Finally, the sample holder was removed from the stand and the sample was thoroughly cleaned and dried.

In order to measure the depth of diffusion, the slide was carefully cut and placed vertically under a microscope. The slide was illuminated from the bottom and the depth of diffusion was measured using the cross wire in the eyepiece and the vernier attached to an optical microscope. The magnification of the microscope ranged from 100 to 400 and in the highest magnification the accuracy of measurement was $\pm 0.5$ $\mu$m. For channel waveguides both depth as well as width were measured simultaneously.

## III. RESULTS AND DISCUSSION

First planar (no mask) diffusion depths were measured for various mole fractions of $AgNO_3$ with $NaNO_3$. The electric field was kept 250 V/cm and all the diffusions were performed at 350 °C for 30 min duration. The silver mole fractions were varied from 0.001 to 0.03. Figure 4 shows the diffusion depth variation for the above range of silver mole fractions. The depth of diffusion showed a monotonic increasing behavior with increasing concentrations of silver. This indicates the existence of a concentration dependent diffusion coefficient. This type of behavior was reported by Ramaswamy and Najafi for their planar waveguides fabricated using the electrolytic releasing silver ion technique.[21] They obtained a depth of 13.5 $\mu$m for a diffusion performed at 300 °C, $t=30$ min, $E=300$ V/cm, and mole fraction 0.002. In the present work the depth gave a value of 23 $\mu$m at 350 °C, $t=30$ min, $E=250$ V/cm, and mole fraction of 0.002. At higher silver concentrations there seems to be saturation trend for depths showing that $Ag^+$ diffusion reaches a solubility limit. The above workers observed the same trend in depth saturation but at a lower silver mole fraction. This may be possible because of the use of different glass samples in both these experiments.

Channel diffusions were performed using masks patterned in aluminum films. Three masks of aperture widths, 10, 50, and 100 $\mu$m were tried. Figure 5 shows the variation of depth and width of diffusion as a function of aperture width of the masks for two time durations. The field was kept at 250 V/cm. Here width is defined as the distance between the edges due to lateral diffusion. Narrow apertures were found to yield deeper penetration and found to have more wider diffusions for a given diffusion time.[2,4,22] 100 $\mu$m aperture showed a bilobed appearance of the diffusion profile as observed in previous studies.[9,10]

Finally, for the aperture width of 10 $\mu$m, the field was varied from 50 to 300 V/cm and the effect on the depths and widths was studied. The diffusion was carried out at 350 °C for silver molar fraction of 0.003. Both showed a linear increase with field as shown in Fig. 6. This shows that greater depths of diffusion could be achieved in a given time by using small aperture masks and increasing the field. Similar results were observed by Forrest *et al.* for their channel waveguides.[9]

Since in the present investigation the emphasis was not on the optical studies, detailed studies were not done on optical properties. But some optical loss studies indicated losses in the range of 0.5–1.0 dB/cm at 632.8 nm wavelength for planar waveguides. The lower loss was again observed for low silver concentrations.[2]


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